1,187,305

PATENT SPECIFICATION

NO DRAWINGS

1,187,305

Inventor: GEORGE STEPHEN FOERSTER

Date of Application (No. 23722/67) and filing Complete Specification: 22 May, 1967.

Complete Specification Published: 8 April, 1970.

Index at acceptance:—C7 A(B249, B25Y, B287, B289, B27X, B271, B273, B275, B277, B279, B289, B309, B319, B339, B34Y, B345, B347, B35Y, B35X, B37Y, B379, B38X, B381, B383, B385, B387, B389, B399, B419, B439, B44Y, B440, B449, B45X, B451, B453, B455, B457, B459, B46Y, B479, B48X, B481, B483, B485, B487, B489, B519, B539, B549, B55Y, B558, B559, B610, B613, B616, B619, B62X, B621, B624, B627, B630, B633, B635, B66X, B661, B663, B665, B667, B669, B670, 741, 742, 743, 744, 770, 781, 782); B3 P(5A, 9, 14)

International Classification:—C 22 f 1/06

COMPLETE SPECIFICATION

Process for production of Extruded Magnesium-Lithium Alloy Articles

We, THE DOW CHEMICAL COMPANY, a Corporation organised and existing under the laws of the State of Delaware, United States of America, of Midland, County of Midland, State of Michigan, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed to be particularly described in and by the following statement:—

The invention relates to a process for production of extruded magnesium-lithium alloy articles having improved strength properties.

Magnesium-lithium alloys have been of interest heretofore but such alloys containing less than about 5 percent lithium have had relatively low strength properties, and alloys containing greater quantities of lithium are uneconomical and tend to overage at room temperatures resulting in loss of initial high strength properties.

It has now been found that additions of lead or tin to magnesium containing lithium in a critical range of proportions produces alloys which when extruded and age hardened can produce articles having unusually high strength values. The present invention accordingly provides a process for the production of such articles, which process is characterized by (a) heating to a temperature within the range of from 300 to 425°C. a magnesium-base alloy consisting, by weight, of from 0.4 to 2.0 percent lithium, from 3 to 20 percent tin or lead or a binary mixture of tin and lead, up to 4 percent aluminium, up to 2 percent manganese, up to 3 percent zinc, up to 0.2 percent zirconium, and the balance magnesium together with such impurities as are normally present therein; (b) die expressing the heated alloy at a rate of at least 15 m. per minute; and (c) hardening the die expressed alloy at a temperature within the range of from 120° to 230°C.

In more preferred ranges of compositions, the alloy contains from 0.4 to 2 percent by weight of lithium and from 3 to 15 percent by weight of tin or from 5 to 20 percent of lead.

The alloy containing lithium and tin or lead may be further improved or modified by the addition of up to 4 percent of aluminum, up to 2 percent of manganese, up to 3 percent of zinc and up to 0.2 percent of zirconium. These additions are generally made to improve mechanical properties and may be made severally, or plurally in any combination. The addition of manganese tends also to improve the corrosion resistance of the alloy. Additions of aluminum and manganese are often made concurrently to the same alloy. More preferred ranges of additions are: up to 2 percent of aluminum, up to 1.5 percent of manganese, up to 1.5 percent of zinc, and up to 0.1 percent of zirconium.

It is to be noted that while tin and lead both improve the properties of the basic magnesium-lithium alloy and both additions produce a metal which is quite advantageously extruded, the alloys containing tin and the alloys containing lead are different in some respects. The alloys containing lead exhibit reasonably good resistance to salt water, which

[*Price 5s. 0d.*]

is surprising on considering the wide spread in solution potential between lead and magnesium. The corrosion resistance of the tin alloys is better than the lead alloys. To attain maximum strength, the alloy containing tin is rapidly cooled as by water quenching the metal immediately after subjecting the metal to a wrought operation such as extrusion.

The alloy may be made in the desired proportions according to the invention by melting together the alloying ongredients in proper proportions or by using hardeners of magnesium alloys containing the alloying constituents. Protection from oxidation during alloying can be effected by the use of a saline flux, as in conventional alloying of magnesium. The molten alloy may be flux refined, if desired, by stirring the alloy with additional flux. The so-refined metal is allowed to settle and then is separated from the flux as by decanting into a suitable casting mold, e.g., a round mold for extrusion stock.

In preparing extruded material, it is desirable first to scalp the cast extrusion stock so as to present a smooth, clean surface to the extrusion die. The clean extrusion stock is heated to a temperature of from 300 to 425°C., inserted into the container of an extrusion press and die expressed. The present alloy is extruded at speeds of at least 15 m. per minute and may even be expressed at speeds of 30 m. per minute or more, substantially without reduction in mechanical strength properties. According to the method of the invention, the present alloys containing tin are advantageously water quenched as the extrusion emerges from the die. The extruded metal, with or without water quenching, is then subjected to an aging step. Aging is carried out by heating the metal at a temperature of 120° to 230°C. until a substantial amount of precipitation hardening takes place. The time of aging will depend on the temperature employed and may vary from about a week at the lower temperatures to about 2 hours at the higher temperatures. Aging at about 175°C. for 24 hours has given excellent results.

The following examples further illustrate the invention.

EXAMPLES

Melts of compositions according to the invention as well as alloys used for comparison were prepared by melting together, under saline flux, magnesium and the individual metallic constituents. The melts were flux refined and settled in a conventional fashion and cast into 75-mm. diameter extrusion billets. The billets were scalped, preheated to about 340°C., inserted in the container of an extrusion press which had been preheated to 315 to 340°C. The metal was pushed into 1/16-inch by 7/8-inch (1.6 x 22 mm.) strip at a rate of 30 m. per minute. Part of each push was water quenched. Samples of quenched and unquenched extruded metal were aged at 175°C. for 24 hours and then subjected to physical testing along with samples of the extrusion which had not been aged. The test results and the compositions prepared and the processing conditions are indicated in the following tables.

In Table I there is illustrated the beneficial effect of adding various amounts of lithium to a magnesium-base alloy containing 5 percent of tin. Note also the beneficial consequences of aging and of water quenching. In Table II there is illustrated the improvement in properties on adding various amounts of tin to a magnesium-lithium alloy containing 1.5 per cent of lithium. In Table III there is illustrated the high level of strengths obtained on making additions of aluminum, manganese, zinc, or, aluminum and manganese to a magnesium-lithium-tin alloy. In Table IV there is illustrated the property levels obtained on adding various amounts of lithium to a magnesium alloy containing 9 percent of lead. In Table V there is illustrated the increased strength properties exhibited on adding increasing amounts of lead to a magnesium-lithium alloy containing 1.5 percent of lithium. In Table VI there is illustrated the effect of adding zirconium or zirconium and zinc to magnesium-lithium-lead alloy.

In the tables, comparative examples outside the scope of the invention are indicated by an asterisk (*). The balance of the alloy compositions is magnesium. Abbreviations used in the tables are as follows:

% E = Percent elongation

TYS = Tensile yield strength in kg./cm.²

TS = Ultimate tensile strength in kg./cm.²

TABLE I

Example No.	Alloy Composition, Wt. %					*Quenched, not Aged			Quenched and Aged			Aged, not Quenched		
	Sn	Li	Al	Mn	Zn	% E	TYS	TS	% E	TYS	TS	% E	TYS	TS
1*	5	0	—	—	—	10	1610	2590	10	1610	2590	—	—	—
2*	5	0.3	—	—	—	6	1890	2730	7	2380	3080	8	2100	2800
3	5	0.8	—	—	—	3	1840	2410	3	3500	3710	4	2870	3220
4	5	1.6	—	—	—	4	1540	2170	2	3290	3500	4	2660	2870
5*	5	3.7	—	—	—	9	1190	1840	4	1890	2410	8	1120	1750

TABLE II

Example No.	Alloy Composition, Wt. %					*Quenched, not Aged			Quenched and Aged			Aged, not Quenched		
	Sn	Li	Al	Mn	Zn	% E	TYS	TS	% E	TYS	TS	% E	TYS	TS
6	3	1.5	—	—	—	4	1470	2030	2	2870	3010	6	2170	2520
7	5	1.5	—	—	—	4	1540	2170	2	3290	3500	4	2660	2870
8	7	1.5	—	—	—	6	1750	2310	2	3080	3360	5	2240	2520
9	9	1.5	—	—	—	5	1890	2520	1	3150	3150	2	2520	2870
10	11	1.5	—	—	—	2	2170	2590	—	—	—	4	2590	3220
11	14	1.5	—	—	—	4	2100	2730	2	3430	3850	4	2660	3150

TABLE III

Example No.	Alloy Composition, Wt. %					*Quenched, not Aged			Quenched and Aged			Aged, not Quenched		
	Sn	Li	Al	Mn	Zn	% E	TYS	TS	% E	TYS	TS	% E	TYS	TS
12	7	1	1	—	—	11	1820	2520	1	3710	3990	6	2940	3290
13	7	1	—	1	—	5	1890	2520	2	3850	3990	4	3220	3500
14	7	1	1	1	—	6	1890	2660	1	3780	3920	1	3360	3500
15	7	1	2	1	—	8	1890	2800	2	3990	4270	3	3430	3570
16	7	1	—	—	1	11	1610	2660	2	3640	3920	4	3150	3570

TABLE IV

Example No.	Alloy Composition, Wt. %				*Quenched, not Aged			Quenched and Aged			Aged, not Quenched		
	Li	Pb	Zr	Zn	% E	TYS	TS	% E	TYS	TS	% E	TYS	TS
17	0.4	9	—	—	12	1960	2660	8	3150	3360	5	3220	3430
18	0.8	9	—	—	6	1540	2450	3	3500	3780	3	3570	3710
19	1.7	9	—	—	6	1470	2170	2	3220	3360	2	3080	3220
20*	3.8	9	—	—	10	1050	1890	4	2030	2450	4	1820	2310
21*	7.5	9	—	—	14	1330	1820	10	1540	1960	11	1190	1750

TABLE V

Example No.	Alloy Composition, Wt. %				*Quenched, not Aged			Quenched and Aged			Aged, not Quenched		
	Li	Pb	Zr	Zn	% E	TYS	TS	% E	TYS	TS	% E	TYS	TS
22	1.5	3	—	—	4	1190	1750	2	2730	2870	2	2660	2870
23	1.5	6	—	—	4	1400	1960	1	3080	3150	2	2940	3080
24	1.5	9	—	—	6	1470	2170	2	3220	3360	2	3080	3220
25	1.5	12	—	—	5	1400	2240	1	3430	3500	1	3360	3430
26	1.5	20	—	—	10	1750	2660	2	3850	4130	—	—	3570

TABLE VI

Example No.	Alloy Composition, Wt. %				*Quenched, not Aged			Quenched and Aged			Aged, not Quenched		
	Li	Pb	Zr	Zn	% E	TYS	TS	% E	TYS	TS	% E	TYS	TS
27	1	9	Trace	—	8	1680	2450	—	3710	3710	—	—	3500
28	1	9	Trace	1	14	1680	2660	1	3850	3990	1	3780	3920
29	1	9	Trace	2	8	1750	2800	—	—	3570	1	3710	3780

Trace = 0.08 — 0.1 percent zirconium as determined spectrophotographically.

WHAT WE CLAIM IS:—

1. Process for the production of extruded magnesium-lithium alloy articles having improved strength properties characterized by (a) heating to a temperature within the range of from 300 to 425°C. a magnesium-base alloy consisting, by weight, of from 0.4 to 2.0 percent lithium, from 3 to 20 percent tin or lead or a binary mixture of tin and lead, up to 4 percent aluminum, up to 2 percent manganese, up to 3 percent zinc, up to 0.2 percent zirconium, and the balance magnesium together with such impurities as are normally present therein; (b) die expressing the heated alloy at a rate of at least 15 m. per minute; and (c) age hardening the die expressed alloy at a temperature within the range of from 120° to 230°C.

2. A process as claimed in Claim 1, wherein the die expressed alloy containing tin is rapidly cooled as it emerges from the press.

3. A process as claimed in Claim 1 or 2, wherein the magnesium-base alloy employed consists, by weight of from 0.4 to 0.2 percent lithium, from 3 to 15 percent tin, up to 2 percent aluminum, up to 1.5 percent manganese, up to 1.5 percent zinc, and the balance magnesium together with such impurities as are normally present therein.

4. A process as claimed in Claim 1, wherein the magnesium-base alloy employed consists, by weight, of from 0.4 to 2.0 percent lithium, from 5 to 20 percent of lead, up to 2 percent aluminum, up to 1.5 percent zinc, up to 0.1 percent zirconium, and the balance magnesium together with such impurities as are normally present therein.

5. A process for the production of extruded magnesium-lithium alloy articles according to Claim 1 substantially as described hereinbefore with reference to any one of the examples.

6. Extruded magnesium-lithium alloy articles whenever produced by the process of any of the preceding claims.

BOULT, WADE & TENNANT,
111 and 112, Hatton Garden, London, E.C.1.,
Chartered Patent Agents.
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1970.
Published by the Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.